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## AFOSR - Final Progress Report

### Surface Microanalysis Investigations of Friction and Lubrication

Grant No. AFOSR-F49620-98-1-0305  
Duration - Mar. 1, 1998 to Feb. 28, 1999

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#### **Summary**

An apparatus has been designed, constructed, and tested that will allow microanalysis of surfaces used in tribological investigations. This is the second generation improvement on a unique apparatus at Carnegie Mellon University that allows measurement of frictional forces between single crystalline metal surface prepared and characterized under ultra-high vacuum conditions. This is based upon a vacuum tribometer that allows simultaneous measurement of normal and shear forces between single crystalline surfaces. Prior work with this apparatus had relied upon preparation and analysis of surfaces prior to their being brought into contact for friction measurements. It has been limited by the inability to identify and analyze microscopic contact points on the surface following friction measurements. The modifications to the apparatus include the addition of a Scanning Electron Microscope and an Scanning Auger Microprobe which allow both imaging and elemental analysis of the surfaces.

## Project Objectives

The primary objective of this project has been the upgrading of equipment used in the P.I.'s lab for fundamental investigations of tribological phenomena. The existing apparatus used in the P.I.'s lab consists of an ultra-high vacuum surface analysis chamber which includes various instrumentation for surface preparation, surface analysis, and measurements of surface frictional properties. A schematic is shown in figure 1. At any point in time the apparatus houses and uses two single crystal metal surfaces which are used to form solid-solid interfaces for friction measurements. For the most part these are single crystalline metal surfaces, both of the same material.

Surface preparation uses standard methods employed in the field of surface science for cleaning of metal single crystal surfaces. Removal of many contaminants is often done using  $\text{Ar}^+$  ion sputtering. This is then followed by annealing to high temperatures ( $\sim 1000\text{K}$ ) in order to obtain well order single crystalline surfaces. Other cleaning methods include high temperature exposure to oxygen. The primary objective of these is to produce surface that are cleaned of contaminants prior to making friction measurements. In experiments in which the effect of surface modification are of interest the surface are then exposed to gas phase species which can adsorb on the surfaces. Prior to friction measurement. These modifications include: the adsorption of molecular species, oxidation by exposure to  $\text{O}_2$  and other treatments.

Analysis of the surface prior to the making of friction measurements makes use of Auger electron spectroscopy (AES), Low Energy Electron Diffraction (LEED), and thermally programmed reaction spectroscopy (TPRS). AES and LEED determine the composition of the surfaces and the crystallographic structure and relative orientations of the two single crystals. TPRS is used to investigate the surface chemistry of adsorbates and to determine the coverages of molecular adsorbates. These are all done to determine the states of the surfaces prior to making friction measurements.

Friction measurements between pairs of single crystalline surfaces are made using an ultra-high vacuum tribometer. This device is unique to the P.I.'s laboratory. Once both single crystalline surfaces have been prepared they are brought into contact under a well defined normal load and then sheared relative to one another. Both the normal forces (load) and the shear force (friction) can be measured simultaneously during

sliding. The device allows variation of: load, sliding speed, temperature, etc. In addition, of course, it allows variation and control of surface properties prior to friction measurement.

A number of interesting tribological problems have been studied with the UHV tribometry apparatus in its configuration prior to this project. These include the effects on metal-metal friction of: adsorbed monolayers, crystallographic orientation, and adsorbate layering. In addition a detailed study of the frictional properties of quasicrystalline surfaces has been performed which measured the intrinsic friction between truly clean quasicrystalline surfaces and the role of surface oxidation on quasicrystal friction. The important capability of the existing apparatus has been its ability to allow measurements of surface characteristics prior to friction measurements. The primary limitation of the existing UHV tribometry apparatus was that following friction measurements it was not possible to perform analysis of the surfaces at the points of contact at which shearing had occurred. This has been remedied by the improvements made possible by the funds supplied by this grant.

### **Technical Progress and Accomplishments**

The apparatus described above has been completely redesigned to include instrumentation that allow surface microanalysis following friction measurements. A schematic of the apparatus in its current form is shown in figure 2. This has required construction of a new UHV chamber to house the additional instrumentation for scanning electron microscopy (SEM) and scanning Auger microscopy (SAM). The additions include a small spot electron gun, a secondary electron detector, and a hemispherical analyzer for Auger electron microscopy. In addition a differentially pumped  $\text{Ar}^+$  ion gun has been purchased in order to improve the operation of the apparatus. The original instrumentation including the tribometer, the mass spectrometer, and the optics for Low energy electron diffractions are also present in the new apparatus.

It has been possible to purchase some instrumentation that is substantially better than that in the original proposal due to some highly competitive bidding on the part of the vendors. The electron gun is made by FEI Components and is model SD 2LE EVA. This has a minimum spot size of  $< 20\text{nm}$  at a beam voltage of 25 keV which is entirely

sufficient for the experiments that we wish to perform. The secondary electron detector (SED) and the hemispherical analyzer were both made by Omicron Associates. The analyzer is a model EA125 U7 which has a mean radius of 125mm and seven channel electron detection. This is a very high sensitivity analyzer which can be used in the future also for x-ray photoelectron spectroscopy with the addition of an x-ray source to the apparatus. The UHV chamber was built by MDC Corp. and was custom designed for the project. In addition to these components some ancillary instrumentation including an infrared spectrometer was purchased with the remaining matching funds provided by the P.I. for this project.

During the period of the grant the new instrument was designed, and the commercially available components order and received. Construction of the new apparatus has been a major undertaking. At this time it is now completed and is in operation in the P.I.'s laboratory. All the components have been tested individually and the students using the apparatus are now trained in the use of the new instrumentation. Using the SEM it has been possible to obtain images of the wear tracks that form on the surfaces of crystals during sliding. The dimensions of the contact points are on the order of 1-5  $\mu\text{m}$  in width and hence the 20nm resolution of the electron beam is quite sufficient to provide images of these wear tracks. At this point in time the SAM capability is being optimize for spatial mapping of the elemental composition of the wear tracks.

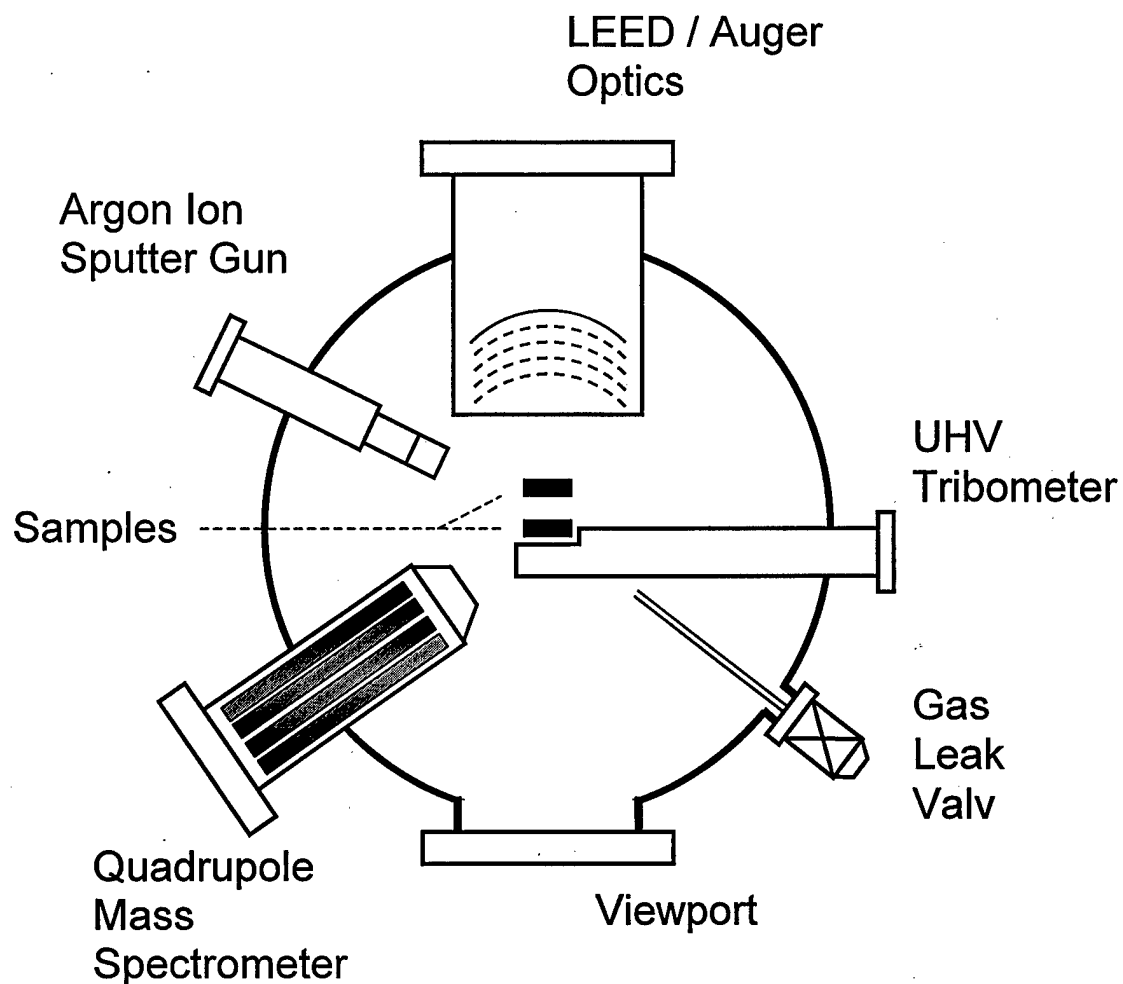


Figure 1. Schematic representation of UHV tribometry chamber prior to modifications made during the course of the DURIP grant period. The schematic shows a cross section through the main operating region of the apparatus. One of the two samples is mounted to the tribometer while the other is free to move within the chamber. (newfigures/projects/Ni friction/ Fig06a).

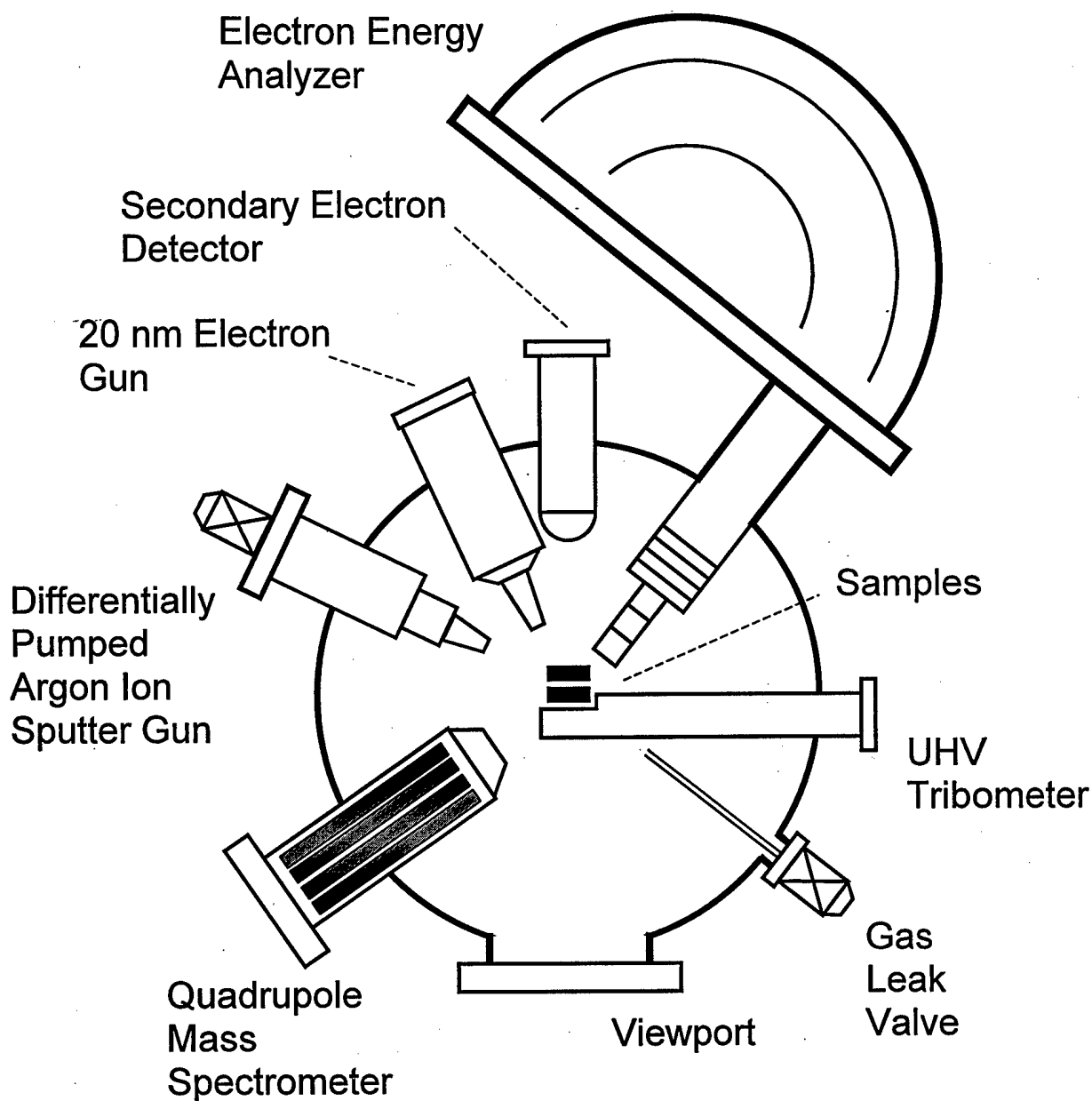


Figure 2. Schematic of the UHV tribometry apparatus after modification made during the DURIP grant period. The low energy electron diffraction (LEED) optics are now mounted on a lower level section of the apparatus. (newfigures/projects/Ni friction/ Fig06b).

### Equipment Purchases

1. FEI High resolution electron gun (FEI model SD 2LE EVA)	\$115,000
2. Electron energy analyzer and secondary electron detector. (Omicron model EA125 U7)	\$ 94,000
3. Magna Infrared Spectrometer	\$ 40,089
4. Cary UV-Vis spectrometer	\$ 21,283
Total	<hr/> \$270,372*

\* Note the excess expenditure over the original budget of \$259,000 was made through use of unrestricted from both the department and from the P.I.